TEST PROCEDURE	TP 113C
Title Density Measurement of Petroleum Fuels	**Page Number** 1 of 15
Originator Carl A. Scarbro	**Supersedes** TP 113B
Responsible Organization Fuels and Chemical Analysis Branch (FCAB)	**Computer Program** TP 113 Database
Type of Test Report Computer	**Data Form Number** Form 113-01
Report Distribution Project Officer and Chemistry Laboratory File	**Implementation Date** 10-16-95

Implementation Approval

Original Test Procedure Authorized by EPCN #143 on 04-18-94

Revision Description

10-16-95 The purpose of this change is to revise the procedure as described in EPCN #193.

Note: Specific brand names in EPA/EOD procedures are for reference only and are not an endorsement of those products.

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Attachments

1. Purpose

This method is the EOD-specific version of ASTM D 1298-85, "The Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method," a method quoted in most EOD fuels procurement, federal vehicle emission, and fuels enforcement test programs.

This method is suitable for natural gasolines, motor gasolines, aviation turbine fuels, special boiling point spirits, naphthas, white spirits, kerosines, gas oils, distillate fuel oils, and similar petroleum products with a Reid Vapor Pressure of 26 psi or less.

2. Test Article Description

Petroleum product sample of 500 mL in volume

3. References

3.1 American Society for Testing and Materials (ASTM) D 1298-85 Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

3.2 Petroleum Measurement Tables, Volume Correction Factors, ASTM D 1250-80

3.3 American Society for Testing and Materials (ASTM) Standard Practice E 177-86, Standard Practice for use of the Terms Precision and Bias in ASTM Test Methods

3.4 ASTM Manual MNL7, Manual on Presentation of Data and Control Chart Analysis: 6th Edition

3.5 American Society for Testing and Materials (ASTM) E 29-90, Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specification

3.6 "FileMaker Pro User's Guide," 1990 Edition

4. Required Equipment

4.1 Hydrometers meeting ASTM specification E 100, Numbers 1H to 10H, long, plain

4.2 Thermometers meeting ASTM specification E 1 No. 12F or equivalent resistance temperature measurement device (RTD) with NIST-traceable calibration

4.3 Fume hood

4.4 250-to 500-mL hydrometer cylinder with an inside diameter at least 25 mm greater than the largest diameter hydrometer used

For mixtures with non-petroleum products, the hydrometer cylinder must be water jacketed and connected to a circulating constant temperature bath.

4.5 Constant temperature bath capable of maintaining 60.0 ±0.5 °F

The constant temperature bath is required for oxygenated samples and is optional for other sample types.

4.6 Ruler with millimeter scale

4.7 Rinse bottles for glassware cleaning

4.8 Reagents

4.8.1 N-pentane [109-66-0] commercial grade

4.8.2 Control Gasoline substantially similar to the emission certification test gasoline specified in 40 CFR 86.113

4.8.3 Control Diesel Fuel substantially similar to the emission certification #2 diesel specified in 40 CFR 86.113

4.8.4 De-ionized distilled water [7732-18-5], bp 100 °C

4.8.5 Micro Liquid Laboratory Cleaner, or equivalent

4.9 Density Work Sheet, Form 113-01 (Attachment A)

4.10 Control Chart for the Range of Duplicate Analyses, Form 109-01 (see TP 109)

4.11 Control Chart for Individual Observations, Form 109-02 (see TP 109)

4.12 Chain of Custody, Form 109-03 (see TP 109)

4.13 Instrument, Fuel-Type Tally Sheet, Form 115-01 (see TP 115)

4.14 Official Sample Seal, EPA Form 7500-2

5. Precautions

5.1 N-pentane and gasoline or other sample materials are hazardous materials. Specific procedures for handling these materials are listed in their respective Material Safety Data Sheets (MSDS) on file in FCAB. The analyst must be familiar with these MSDS procedures prior to performing this procedure.

5.2 In case of a spill of any of the above materials, remove all personnel from the area, contact the Emergency Response Team, and, if possible, turn the equipment off.

Spills of a gallon or less can be mopped up with spill control pillows or absorbent. Collected materials must be disposed of according to Federal and local waste disposal rules.

5.3 Samples, standards, and glassware must be at the prescribed test temperatures for their fuel group to ensure test accuracy. Prior to testing, samples, hydrometer, thermometers, and hydrometer cylinders should be stored in the same environment in which they will be tested. Generally this will be a fume hood at room temperature for diesel fuels, and a constant temperature bath for gasoline and oxygenated gasoline.

5.4 All glassware must be free of any cracks and be thoroughly clean to the eye.

6. Visual Inspection

6.1 Samples are inspected before analysis for phase separation, leaks, and tampering.

6.2 Glassware is inspected to make sure it is clean and dry.

6.3 RTD temperature readout is checked to make sure that it has a current calibration sticker.

7. Test Article Preparation

7.1 Obtain and inspect samples as described in the FCAB Enforcement Sample Chain-of-Custody Procedure in TP 109. Note any unusual conditions on Form 113-01.

7.2 Refer to the following table to identify the test conditions that meet the fuel group to be tested. Bring the temperature of the samples, quality control materials, hydrometer, hydrometer cylinder, and thermometer or RTD into the temperature range outlined in the following table.

Operating Conditions

Sample Type	Initial Boiling Point	Test Temperature
Highly Volatile	NA	Cool in original container to <35 °F
Moderately Volatile (gasolines)	≤250 °F	Cool in original container to <65 °F
Moderately Volatile and Viscous	≤250 °F	Heat to minimum temperature to obtain sufficient fluidity
Nonvolatile (diesel fuels)	≤250 °F	Any temperature between 0 °F and 195 °F
Mixtures with Non-petroleum Products (oxygenated gasolines)	NA	Test at 60 ±0.5 °F

7.3 Plan the analysis run. Refer to Form 115-01 to determine which quality control samples need to be analyzed with the current sample batch.

7.3.1 Provide for at least one laboratory duplicate per 20 analyses per hydrometer.

7.3.2 A control gasoline or control diesel fuel must be analyzed at least once per every 20 analyses per hydrometer. The choice of gasoline versus diesel control fuel will be made on the relative number of samples per fuel group analyzed.

8. Test Procedure

101 On Form 113-01, record the sample ID, hydrometer serial number and thermometer or RTD serial number.

102 Transfer the sample to a clean hydrometer cylinder. Do not allow the sample to splash. This will minimize the formation of air bubbles and the evaporation of the lower-boiling constituents of volatile samples. Transfer gasolines and other volatile samples by siphoning.

Remove any air bubbles on the surface of the sample by touching them with a piece of clean filter paper before inserting the hydrometer. There must be sufficient sample in the cylinder so that the hydrometer will float at least 25 mm above the bottom.

Oxygenated fuels or other mixtures containing non-petroleum products must be analyzed in a jacketed hydrometer cylinder connected to a constant temperature bath set at 60 °F.

103 Place the cylinder in a vertical position free of air currents. The temperature of the surrounding medium, the air or the constant temperature bath, must not change by more than 5 °F over the course of the test. Monitor this temperature periodically during the analysis, and take steps to stabilize the temperature if required.

104 Lower the hydrometer gently into the sample. Take care to avoid wetting the stem above the level to which it will be immersed in the liquid.

105 Verify that the hydrometer is at least 25 mm above the bottom of the cylinder.

106 Continuously stir the sample with the thermometer, RTD, or a stirring rod. Take care that the mercury thread or RTD tip is kept fully immersed, and that the stem of the hydrometer is not wetted above the immersion level.

107 As soon as a steady reading (less than 0.5 °F temperature change) is maintained for 1 minute, record the sample temperature to the nearest 0.5 °F on Form 113-01 as the "Initial Temp (°F)." For mixtures with non-petroleum products, the initial sample temperature must be 60.0 ±0.5 °F.

108 Depress the hydrometer about two scale divisions into the liquid, and then release it. The remainder of the stem of the hydrometer must be kept dry since unnecessary liquid on the hydrometer affects the reading. With samples of low viscosity, impart a slight spin on the hydrometer. Allow sufficient time for the hydrometer to come to rest, and for all air bubbles to come to the surface. The hydrometer must float without contacting the cylinder walls.

109 When the hydrometer has come to rest, floating freely away from the walls of the cylinder, estimate the hydrometer reading to the nearest 0.05 °API. The correct hydrometer reading is that point on the hydrometer scale at which the principal surface of the liquid cuts the scale, not the meniscus about the surface of the hydrometer. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale. On Form 113-01, record the hydrometer reading as the "Observed API Gravity."

110 With an opaque liquid, take a reading by observing, with the eye slightly above the plane of the surface of the liquid, the point on the hydrometer scale to which the sample rises. This reading, at the top of the meniscus, requires correction since the hydrometers are calibrated to read at the principal surface of the liquid. The correction for the particular hydrometers in use may be determined by subtracting 0.1 °API from where the top of the meniscus cuts the hydrometer scale.

On Form 113-01, note that the reading was opacity corrected.

111 Immediately after observing the hydrometer scale value, again cautiously stir the sample with the thermometer, RTD, or stirring rod. Keep the mercury thread or RTD tip fully immersed. On Form 113-01, record the temperature as the "Final Temp (°F)." Should this temperature differ from the previous reading by more than 1 °F, repeat the hydrometer test and then the thermometer observation until the temperature becomes stable within 1 °F.

112 Verify, and reset if necessary, the date and time settings on the computer that contains the file "TP 113 Database."

113 Open the file "TP 113 Database." The input screen will automatically come up on the screen (see Attachment B). Append a new record and enter the sample ID and the analyst's initials in the appropriate fields. Enter from TP 113-01 the "Final Temp (°F)" and the "Observed API Gravity" in their respective fields.

Note: The computer will compute the "Truncated API" and the "Rounded API." Truncated API is defined as the observed API gravity, which is recorded on Form 113-01, rounded down to the nearest 0.5 °API. Rounded API" is defined as the observed API gravity rounded up to the nearest 0.5 °API.

114 Open "Petroleum Measurement Tables, Volume Correction Factors," Volume II, Table 5B. Enter the table in the column labeled "API Gravity at Observed Temperature" equal to the "Truncated API" shown on the input screen.

Follow that column down until it is cross referenced with the temperature that equals that of the "Final Temp (°F)" on Form 113-01. Enter the value that lies at that intersection in the field labeled "Truncated API @ 60 °F."

Reenter the table in the column "API Gravity at Observed Temperature" with "Rounded API" shown on the input screen. Follow that column down until it is cross referenced with the temperature that equals that of the "Final Temp (°F)" on Form 113-01. Enter the value that lies at that intersection in the field labeled "Rounded API @ 60 °F."

The final correction is calculated by the computer using the following equation:

Degrees API @ °F = B+[[(A-B)/(D-E)]*(C-E)], where:

A = Truncated API @ 60 °F
B = Rounded API @ 60 °F
C = Observed API Gravity
D = Truncated API
E = Rounded API

115 If the “Observed API Gravity” was opacity corrected, put a note to that effect in the “Comments” field of the input screen. Transcribe any other unusual conditions noted on Form 113-01 to the “Comments” field of the input screen.

116 Enter an X on the appropriate Form 115-01 for each successful analysis.

117 Pour or drain the sample into a waste container designated for the sample product, gasoline with gasoline and fuel oil with fuel oil. Rinse the cylinder and hydrometer with n-pentane and pour the rinse into a gasoline waste container. Air dry both the hydrometer and cylinder.

If residues of any sort are seen on the hydrometer or cylinder, rinse with a working solution of 18 mL Micro Liquid Laboratory Cleaner in 1 liter of de-ionized water. Rinse with deionized water and then acetone. Air dry again.

118 Analyze the “Control Gasoline” or “Control Diesel Fuel” in the same manner as the samples. Record the date and the result on the Form 109-02 specific to that standard. Calculate the moving range and plot both the result and the moving range values. Review the control chart for out-of-control indications. Out-of-control indications require immediate corrective action. Corrective action must conclude with successful repetition of the control fluid analysis to verify re-establishment of process control.

If out-of-control occurrences can not be reconciled, consult the laboratory supervisor. Repeat the analysis of any samples identified as being affected by the cause of the out-of-control indication and discard the original results from those samples. Record the results of the investigation and the corrective action taken on the control chart.

119 Record laboratory duplicate values, as they become available, on Form 109-01. Calculate and plot the range. Review the control chart for out-of-control indications. Out-of-control indications require immediate corrective action to resolve any causes of excessive variability. Corrective action must conclude with successful repetition of the duplicate analyses to verify re-establishment of process control.

If out-of-control occurrences can not be reconciled, consult the laboratory supervisor. Repeat the analysis of any samples that are identified as being affected by the cause of the out-of-control indication and discard the original results from those samples. Record the results of the investigation and the corrective action taken on the control chart.

120 After the last sample analysis is completed, print the TP 113 Database Report (see Attachment C) for the current day’s analyses.

9. Data Input

9.1 The “Sample ID,” “Hydrometer Serial #,” “Thermometer Serial #,” “Initial Temp (°F),” “Observed API Gravity,” and “Final Temp (°F)” are recorded by the analyst on Form 113-01.

9.2 The “Sample ID,” “Observed API Gravity,” “Final Temp (°F),” and the analyst’s initials are entered in the TP 113 Database.

9.3 The “Truncated API @ 60 °F” and the “Rounded API @ 60 °F” are identified from the “Petroleum Measurement Tables, Volume Correction Factors,” Volume II, Table 5B and then entered in the TP 113 Database.

9.4 The analysis date (Analyst_D), “Truncated API,” “Rounded API,” “Specific Gravity @ 60/60,” and “Degrees API @ °F” are computed and automatically recorded by the TP 113 Database.

9.5 A Control Chart for the “Range of Duplicate Analyses” (Form 109-01) is used to record duplicate results.

9.6 A Control Chart for Individual Observations (Form 109-02) is used to record Control Gasoline or Control Diesel Fuel results.

9.7 If a hydrometer reading is corrected for sample opacity, the correction is noted on Form 113-01 and in the TP 113 Database.

9.8 A mark is made on Form 115-01 for each sample analyzed in order to keep track of when quality control samples need to be included in the analysis.

10. Data Analysis

10.1 The analyst compares Form 113-01 and the TP 113 Database Report and reviews them for reasonableness, completeness, and conformance to the acceptance criteria. When all corrections are made, the analyst signs and dates Form 113-01.

10.2 A technician reviews Form 113-01 and the TP 113 Database Report to confirm compliance with the acceptance criteria and to ensure that no transcription errors have been made. The technician signs and dates the TP 113 Database Report indicating the validation has been completed.

11. Data Output

11.1 The completed Form 113-01, TP 113 Database Report, and control chart Forms 109-01 and 109-02 are filed according to FCAB Enforcement Sample Chain-of-Custody Procedure.

11.2 Form 115 -01 is stored with the control charts for this procedure.

12. Acceptance Criteria

12.1 The test conditions specified in the table under Section 7.2 are met.

12.2 The difference between the initial and final temperature readings for the hydrometer test must be no greater than 1 °F.

12.3 The temperature of the surrounding media, air or constant temperature bath, must not vary by more than 5.0 °F.

12.4 The hydrometer must float freely in the cylinder at least 25 mm above the bottom.

12.5 The final temperature of mixtures with non-petroleum products must be 60.0 ±0.5 °F.

12.6 A Control Gasoline or Control Diesel Fuel must be analyzed once per every 20 analyses and the results recorded on a control chart, Form 109-02. The values for those fluids must be within the control limits.

If not, an investigation for cause must be conducted and documented on the control chart . Re-establishment of process control must be demonstrated and affected samples must be reanalyzed before further analyses can be performed.

12.7 Laboratory duplicates must be analyzed once per every 20 analyses per hydrometer. Laboratory duplicate range values must be within the control limits.

If not, an investigation for cause must be conducted and documented on the control chart . Re-establishment of process control must be demonstrated and affected samples must be reanalyzed before further analyses can be performed.

13. Quality Provisions

13.1 Laboratory correlation and precision statistics will be reported by FCAB in an annual quality assurance report.

13.2 Repeatability for this procedure has been statistically determined using the control gasoline and the control diesel fuel.

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the normal and current operations of the test method, exceed the following values only in 1 case out of 20:

Gasoline	0.28 °API
#2 Diesel	0.09 °API

13.3 The results of laboratory duplicates and process control fluid measurements are analyzed using statistical process control methods.

13.4 Validation of all analysis data is done by an independent technician.

13.5 Analysis samples sent to other facilities are re-sealed with a new Official Sample Seal, EPA Form 7500-2, and shipped with Form 109-03 to assure sample integrity.

Density Work Sheet

Sample ID.	Hydrometer Serial #	RTD or Thermometer Serial #	Initial Temp (°F)	Observed API Gravity	Final Temp (°F)

Comments:

_____

_____

I have performed this analysis in accordance with the requirements of TP 113.

Name: _____ Date: _____

TP 113 Density Measurement Data Input Screen

FFNumber _____

Sample ID _____

Analysis_D _____ Analyst _____

Observed API Gravity _____ Final Temp °F _____

Truncated API _____ Rounded API _____

Truncated API @ 60 °F _____ Rounded API @ 60 °F _____

Degrees API @ 60 °F _____ Specific Gravity @ 60/60 °F _____

Comments _____

Date Printed 9/27/95

TP 113 Database Report
Fuel Density Measurement

Page 1

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

Analyst: ________ **Analysis Date:** ____________
Sample ID: ________________________________
Specific Gravity @ 60/60 °F: ____________
Density °API @ 60 °F: ____________
Observed °API: ____________
Observed Temp °F: ____________
Look-up Data: Rounded °API @ 60 °F: ____________
(Rounded °API: ____________)
Truncated °API @ 60 °F: ____________
(Truncated °API: ____________)
Comments:

This data has been validated according to TP 113

Validator's Signature: ___________________________________ Date: _______________